

BSA 03-01

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl.No.	:	10/622,843
Applicant	;	Wiesmann et al.
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Title	:	Fluorinated Precursors of
		Superconducting Ceramics, and
		Methods of Making the Same
TC/A.U.	:	1762
Examiner	;	Brian K. Talbot
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Commissioner for Patents P.O. Box 1450 Alexandria, Virginia 22313-1450 I hereby certify this correspondence is being deposited with the United States Postal Service as first class mail, postpaid in an envelope, addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginiq 22313-1450 on May 7, 2007

Signed: 5-7.7

## **DECLARATION UNDER 37 C.F.R. § 1.132**

The undersigned, Dr. Harold Wiesmann of Stony Brook, New York, herewith declares as follows:

- 1. My résumé is attached.
- 2. I am co-inventor of the patent application identified above.
- 3. Our invention includes a method of making fluorinated precursors of superconducting ceramics. The steps of the method include placing a precursor solution onto a substrate to provide a precursor-covered substrate. The precursor-covered substrate is then fluorinated by heating in an atmosphere containing at least one hydrofluorocarbon gas. A fluorinated precursor is formed.

- 4. After formation of the fluorinated precursor, the precursor of our invention can be converted into a crystalline superconductor by annealing. Fluorine in the precursor enhances epitaxial growth during the conversion.
- 5. The crystalline superconductor formed by our invention contains only trace amounts of fluorine.
- 6. Before and after fluorine treatment, the precursors of our invention are substantially non-superconducting.
- 7. In the method described in *Ovshinsky* (U.S. Patent No. 5,520,953), a precursor is treated to provide a material with "at least one superconducting phase." A "material with at least one superconducting phase" is a "superconductor." In the relevant art, it is not appropriate to refer to a "superconductor" as a "precursor."
- 8. The treatment of Ovshinsky which converts the precursor to a superconductor is exposure to a high temperature oxidizing environment.
- 9. In col. 9, line 54; col. 11, line 66; and col. 15, line 24, of Ovshinsky, the term "precursor" is used in reference to the material after its exposure to a high temperature oxidizing environment. Thus, the material is a superconductor at such point. Accordingly, it was inappropriate to use the term "precursor" in these sections.
- 10. The Ovshinsky superconductor is then exposed to fluorine. Accordingly, in the method of Ovshinsky, a superconducting material is fluorinated (i.e., not a precursor).

- 11. The Ovshinsky end product superconductor contains fluorine and is described as a "fluoro-oxide superconducting material." That is, fluorine is incorporated into the structure of the Ovshinsky superconductor. In contrast, the superconductors of our invention contain only negligible amounts of fluorine.
- 12. In one aspect, deBarbadillo (U.S. Patent No.4,962,085) teach simultaneously fluorinating and oxidizing a precursor to prepare fluoridized oxidic superconductors. In this aspect, a fluorinated precursor is not formed.
- 13. In another aspect, deBarbadillo describe a precursor doped with fluorine. The fluorine is added as a solid, i.e., not as a gas. The technique used is "mechanical alloying." In this technique, fluorine is added as a metal fluoride, such as cuprous fluoride powder. The addition of fluorine as a solid results in a material that is physically different from our material. In our invention, the exposure to fluorine gas results in a microscopic distribution of fluorine in the precursor. Thus, we provide a substantially homogenous precursor material. Adding fluorine as a solid per deBarbadillo, the fluorine does not come into intimate contact with the material. Thus, there is no microscopic distribution of the fluorine in the deBarbadillo precursor material.
- 14. In both deBarbadillo aspects, fluorine is structurally incorporated into the superconducting end product. In contrast, the superconductors of our invention contain only negligible amounts of fluorine.
- The gases used in our invention are uniquely successful because each gaseous molecule comprises fluorine, carbon and hydrogen. These gases are basically non-corrosive, non-toxic and some (HFC 134a) are readily available at low cost. None of the cited prior art references teach such gases. At the time of our invention, it would not have been expected that such gases would be suitable for making fluorinated precursors.

- 16. In one embodiment, our invention includes a method of inhibiting the conversion of a fluorinated precursor film into a crystalline superconducting film. The method comprises adding a small amount of fluorinated gas during a heat treatment process by which a precursor film is to be converted into a crystalline film. As described in Example 5: "It was observed that approximately 10 milliTorr of HFC 134a gas was sufficient to stop the conversion of the precursor film into crystalline YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>"; and further "Higher HFC 134a gas pressures were sufficient to convert crystalline YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> back into a precursor like film." *Incredibly*, the crystalline YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> films, made from the fluorinated substantially non-superconducting precursors, can be converted back into a fluorinated substantially non-superconducting precursor. We believe this is due to the fact that synthesized crystalline YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> is exposed to gaseous molecules comprising fluorine and hydrogen. None of the cited prior art describes reversion back to a substantially non-superconducting precursor.
- I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. Further that these statements were made with the knowledge that willfully false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code, and that such willfully false statements may jeopardize the validity of the application of any patent issued thereon.

Respectfully submitted,

Dated:

Harold Wiesmann, Ph.D.

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